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Filtration of Ground Water Samples for Metals Analysis

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ABSTRACT

The filtration of a ground water samples with 0.45 um filters for determination of 'dissolved' metals is not only inaccurate for distinguishing between dissolved and particulate phases, but if used for estimates of mobile contaminant loading in a given aquifer, may result in significant underestimations.

INTRODUCTION

The attachment or incorporation of contaminants to suspended particulates has been proposed as a possible transport mechanism (1,2.3) in ground water. It is evident that such processes occur and can facilitate the transport of inorganic and organic contaminants (1,4,5); however it remains to be determined how significant such transport can be in specific hydrogeological settings.

In the process of characterizing hazardous waste sites, investigators often collect both filtered and unfiltered samples for inorganic analyses. Typically, there are significant (>10%) differences for many elements. Inclusion of particulates bias concentrations high especially for such species as aluminum, silicon, iron and manganese. But when large differences occur for species such as lead and chromium, it is incumbent upon investigators to question their source and address the possibility of facilitated transport of contaminants on mobile colloids.

Analytical methods used to determine "dissolved" metal concentrations have historically used 0.45 um filters to arbitrarily separate dissolved and particulate phases. During use, filters typically become increasingly clogged, reducing the nominal pore size (6,7), further reducing the cutoff for dissolved versus particulate. If the purpose of such determinations is an evaluation of 'mobile' species in solution, significant underestimations of mobility may result. Metal contaminants may move through fractured and porous media not only as dissolved species, but also as precipitated phases, polymeric species or be adsorbed to inorganic or organic particles of colloidal dimensions. Colloidal particles as defined by Stuum (8) have an upper limit of 10 microns (10^{-6} meters) in size. Studies by Yao et al. (9) and O'Melia (10) indicate that colloidal particles in the range 0.1 to 1.0 micron may be most mobile in a sandy, porous medium. Reynolds (11) using carboxylated polystyrene beads ranging from 0.10 to 0.91 um in size, recovered 45% of the 0.91 um size beads, and greater than 70% of 0.10 and 0.28 um size beads in laboratory sand column effluent. Champlin and Eichholz (12) showed that the movement of radioactive sodium and ruthenium in sand beds was associated with particulate matter of micron dimensions.

On the other hand, if the purpose of such filtration is to determine truly dissolved aqueous species, the passage of colloidal material less than 0.45 um in size may result in the overestimation of dissolved concentrations. Hem and Roberson (13) found that some aluminum compounds can pass through a 0.45 um filter yet be retained on a 0.10 um filter. Bergseth (14) found that the measured quantities of Al, Na,

K, Mg and Ca were strongly dependant upon the size and type of filter used and filtrate treatment. Aluminum contents in filtrate increased by as much as a factor of 2 going from 0.05 to 0.45 um filters. In a study by Kim et al. (15), the majority of the concentrations of rare earth elements were found as colloidal species which had passed a 0.45 um filter. Aluminum values differed by a factor of two. Kennedy and Zellweger (16) found errors of an order of magnitude or more in the determination of dissolved concentrations of aluminum, iron, manganese and titanium using 0.45 um filtration as an operational definition for "dissolved".

In addition to the inherent problems of filter type and size, the actual process of collection and filtration of anoxic ground water samples is very difficult without iron oxidation and colloid formation with subsequent sorption of other metals, causing a removal of previously dissolved species to be filtered.

In view of the above, there is concern that the filtration of ground water samples for metal analyses using 0.45 um filters may not provide accurate information concerning the mobility or aqueous geochemistry of metal contaminants. Mobile species may be removed by filtration prior to chemical analysis and other particulate associated metals may pass through such filters and be included as dissolved.

California Superfund Site

At a superfund site in California, filtered (0.45 um) and unfiltered samples were collected from the same wells on the same day. Data for lead is displayed in Figure 1. Differences ranged from 20 to 600 times greater in the unfiltered versus the filtered.

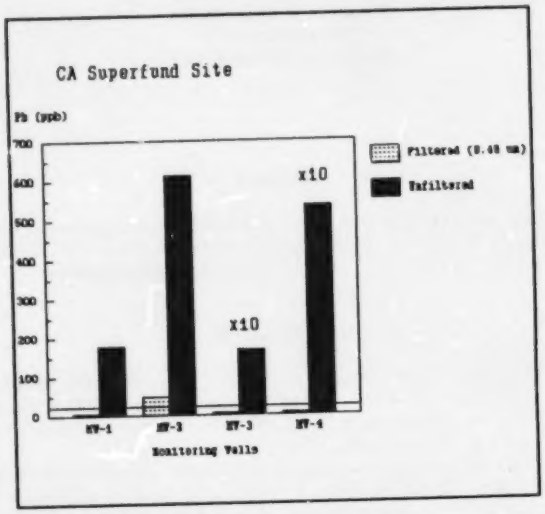

Figure 1 Filtered and Unfiltered Lead Data (horizontal line = maximum contaminant concentration level for drinking water, EPA).

Likewise for chromium (Figure 2), differences were 6 to 24 times greater for the unfiltered. In most samples the differences also fell on either side of the maximum contaminant levels set for drinking water by the Environmental Protection Agency. Such disparities need to be resolved before making decisions about remediation and risk assessment. If sampling artifacts are responsible for the high unfiltered numbers, the source of these artifacts needs to be identified and resolved. Additional filtration work and a review of well construction techniques and materials is needed to determine what portion of the unfiltered concentrations can be attributed to artifacts and what portion may be attributed to mobile colloids.

SAMPLING ARTIFACTS VS. "REAL" GROUND WATER ENVIRONMENT

The disturbance of the subsurface environment as a result of well construction and sampling procedures presents serious obstacles to the interpretation of ground water quality results. Some degree of disturbance

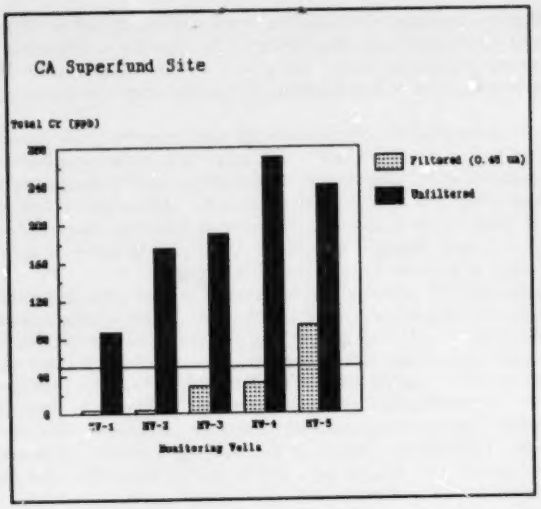

Figure 2 Filtered and Unfiltered Chromium Data (horizontal line = maximum contamination level for drinking water, EPA).

of natural conditions is inevitable. However, the impact of improper well construction and sampling techniques can permanently bias the usefulness and integrity of wells as sampling points. Several aspects of well construction and sampling procedures must be carefully considered to avoid errors associated with the introduction of foreign particles or the alteration of ambient subsurface conditions which may affect natural dissolved or suspended materials.

Well construction

The design, drilling, and construction of monitoring wells have been identified as particularly important steps in the collection of representative water chemistry and hydrologic data. Several references have emphasized the minimization of both the disturbance and the introduction of foreign materials (17,18) because of the potential impact on water chemistry. The RCRA Technical Enforcement Guidance Document (19) suggests that the well must allow for sufficient ground-water flow for sampling, minimize passage of formation materials into the well, and exhibit sufficient structural integrity to prevent collapse of the intake structure. It should be recognized, however, that the well must first provide a representative hydraulic connection to the geologic formation of interest. Without the assurance of this hydraulic integrity, the water chemistry information cannot be interpreted in relation to the dynamics of the flow system or the transport of chemical constituents.

More specific guidance is therefore necessary to maintain or restore the natural hydraulic conductivity of the formation in the vicinity of the screened portion of monitoring wells through the drilling, construction and development procedures. The literature on water well technology can be most helpful in this regard since minimal disturbances of the subsurface is a common goal in maximizing both the yield of water supply wells and the representativeness of water samples and hydraulic information from monitoring wells (20).

To insure the long-term integrity of monitoring wells, particularly with respect to excluding foreign particles and permitting the passage of mobile (i.e., dissolved and suspended) contaminants, specific items which should be observed are:

1) If no alternative to the use of drilling muds or fluids exists, these materials must be removed from the well bore and adjacent formations by careful well development (20). This guidance also applies to the removal of the low permeability "skin" which is caused by abrasion, oxidation and invasive muds which may seal the well bore from the screened interval and bias in situ determinations of hydraulic conductivity (21,22,23). Pumping rates during development should be documented and care should be taken not to exceed these rates during purging or sampling since further development and well damage may aggravate suspended particulate and turbidity problems even in properly designed wells.
2) The emplacement of grouts and seals to isolate the screened interval must be carefully done. The use

of tremie pipes and frequent checking of the depth of emplacement of clay or cement grouts during well construction are strongly encouraged. It is also important to take care to follow manufacturers' guidelines on the hydration of cement or expanding cement as grouts or seals. Excess water addition and grading of cement components or materials due to free fall through standing water can permanently damage the well's integrity (24).

3) Casing and screen materials must be selected to retain their integrity in the subsurface environment (i.e. avoid iron, steel), minimize bias to water samples and insure that screen openings are not reduced by the buildup of corrosion products or by compression. These effects can be checked by repeat determinations of in situ hydraulic conductivity over the useful life of the well. Redevelopment and replacement of the well should be considered if deterioration or significant changes in hydraulic conductivity are observed. Erratic water level readings and sudden changes in turbidity or purging behavior of monitoring wells prior to sampling are warning signs of possible loss of material integrity.

4) Well design fundamentals with regard to the selection of a filter pack and screen size are among the most important issues in obtaining representative hydraulic and water quality information. The exclusion of fines, clays, and silts can be achieved by selecting the grain-size distribution for the filter pack by multiplying the 50-percent retained size of the finest formation sample by a factor of two (Driscoll, 1986). The filter pack material should be cleaned and washed free of fines to insure that extraneous contaminants or particles are removed. The well screen slot openings should be chosen to retain 90% of the filter pack material after development. In natural packed wells it may be advisable to select a screen slot size which will retain at least 50% of the finest material in the screened interval. Minimizing slot screen width however, often leads to greater time and energy spent in well development. The need to document well development procedures cannot be overemphasized.

Maintenance of the hydraulic performance of monitoring wells and the connection of wells to the zones of greatest hydraulic conductivity where contaminant transport is most probable, should take equal importance to the collection of representative water quality data.

Purging and Sampling

Water that remains in the well casing between sampling periods is unrepresentative of water in the formation opposite the screened interval. It must be removed by purging or isolated from the collected sample by a packer arrangement prior to the collection of representative water samples. Water level readings must be made carefully to avoid the disturbance of fines or precipitates which may enter or form in the well due to chemical reactions or microbial processes and accumulate on the interior walls of the well casing screen or at the bottom of the well. Similarly, it is important to purge the stagnant water at flow rates below those used in development to avoid further development, well damage or the disturbance of accumulated corrosion or reaction products in the well. The use of certain sampling devices, particularly bailers and air-lift arrangements, should be discouraged in order to avoid the entrainment of suspended materials which are not representative of mobile chemical constituents in the formation of interest.

A note of caution should be voiced to encourage repetitive sampling of monitoring wells prior to judging the representativeness of determinations of hydraulic conductivity water level readings and water quality data. The effects of the inevitable "trauma" due to drilling, sealing and development of monitoring wells can bias observations of water chemistry until the subsurface is allowed to equilibrate sufficiently (Walker, 1983). Estimates of the time to achieve equilibration vary substantially, particularly when drilling fluids are used in highly permeable formations (20,26); however periods of weeks to several months may be necessary before even major ionic constituents of ground water equilibrate to previous levels (27).

In general, the zone of interest must be isolated, the sample pumped slowly to minimize turbidity and samples collected in such manner as to eliminate O_2 and CO_2 exchange with the atmosphere. No filtration or use of a large pore-size filter (5.0 um) for mobile metals determination is recommended. If desired, additional sampling using in-line filtration with the smallest practical pore size filter may be used for obtaining data for use in geochemical speciation models.

Isolation of Sampling Zone

Isolation of the sampling zone is necessary to minimize the purge volume as well as to minimize air contact. This is especially important since Eh/pH conditions of the formation waters are notoriously sensitive to dissolved gases content. Inflatable packers can be used to achieve isolation of the sampling zone.

Pumping for Sample Collection

It is recommended that a positive displacement pump be used. Other types of sample collection (e.g. bailing)

may cause displacement of non-mobile particles or significantly alter ground water chemistry leading to colloid formation (e.g. vacuum pumps). Surging must be avoided, and a flow rate as close to the actual ground water flow rate should be employed. While an initial approximation, flow rates around 100 to 500 ml/min have been used to successfully sample ground waters in a quiescent mode.

Assessment of Water Constituents While Sampling

Monitoring for dissolved oxygen, temperature, conductivity pH and turbidity during purging and sampling is recommended to determine baseline ground water quality conditions.

Filtration

For estimates of contaminant mobility, filtration with 5.0 um filters using the same procedures as above or collection of unfiltered samples is recommended. Filtration for accurate estimations of geochemistry should be performed in the field with in-line pressure filtration using a large (e.g. 142 mm) polycarbonate-type (thin with sharp pore-size cutoff) 0.1 um filter. Air contact should be minimized and entirely excluded for some samples. Acidification of samples to < pH 2 should be performed immediately. The filter holder should be non-metallic. Holders made of steel are subject to corrosion and may introduce non-formation metals into samples. Prewashing of filters should be routinely performed.

ARIZONA SITE - MATERIALS AND METHODS

To determine the practicality and effectiveness of the above recommendations, research was initiated at a copper mining site in Arizona. The site is located at Pinal Creek in Globe, Arizona about 130 km east of Phoenix and about 170 km north of Tucson. Acidic copper mining wastes have been leaching through the soils and into the ground water at the site for several decades. The geology of the site consists of about 50 m of heterogeneous alluvium overlying Gila conglomerate. Ground water at the site contains high concentrations of dissolved metals and sulfate with pH ranging from 3.6 near the source to pH 7.6 where ground water emerges to the surface and becomes a perennial stream.

A bladder pump (0.6 l/min) and a submersible pump (100 l/min) were used to collect ground water samples from six wells at the site. The bladder pump was used in an attempt to employ a pumping rate which approximated the actual ground water flow velocity. Conservative estimates of ground water flow velocity pass the screened interval were 1.0 l/min and 0.2 l/min for the alluvium and conglomerate, respectively. Purge time was determined by monitoring dissolved oxygen, pH, temperature, conductivity and oxidation-reduction potential (Pt electrode), using a multiparameter field meter equipped with flow-through cell. Sampling was initiated after all readings had reached steady-state values. These values are listed Table 1.

Samples were collected both in air and under nitrogen using a field glove box. Unfiltered and filtered samples were collected using filters ranging in size from 0.03 to 10.0 microns. Samples were acidified in the field with double distilled concentrated nitric acid to pH < 2.

TABLE 1. Ground water quality parameters for wells sampled.

Wells:	MP1W1	X1W4	X1W5	X3W3	X4W3	X5W3
pH	3.71	3.92	6.08	4.27	5.05	5.74
EC(uS)	8500	3020	4300	3210	3200	3620
T(°C)	18.0	18.0	19.0	19.0	18.8	18.9
DO(mg/l)	-	0.14	0.22	0.39	0.24	0.07

RESULTS

Filtration

While it generally took longer to collect the samples, the use of the bladder pump and longer purge

times prior to sampling were not excessive. Filtration within the glove box was difficult and uncomfortable (June temperatures >100°C), especially when trying to handle thin-film polycarbonate filters through gloves. Two wells showed differences (>10%) in some elemental concentrations using different filter sizes. Differences in aluminum, iron and manganese were most notable. Table 2 lists concentrations by ICP for well X3W3. There was a steadily increasing pattern of elemental concentrations using 0.10, 0.40 and 10.0 um filters.

TABLE 2. Elemental Analysis (ICP) for well X3W3 using 0.10, 0.40 and 10.0 micron filters (ppm).

Element	0.1 um	0.4 um	10 um
K	4.73	5.49	9.76
Ca	391	424	492
Mg	91	100	120
Fe	171	187	211
Mn	37.7	40.8	45.5
Al	6.74	7.61	9.93
Ni	0.68	0.75	0.88
Co	0.68	0.75	0.86
Cu	15.0	16.7	19.2
Zn	2.75	3.27	4.13

Similar trends were observed with the submersible pump in well X5W3 (Table 3). These two wells had higher pH than some of the other wells sampled and differences were attributable to a majority of the metal fraction present as dissolved in the pH < 4 wells and greater particle loading at the higher pH sites. Filtered particles accounting for differences were identified with scanning electron microscopy with energy dispersive x-ray and included iron oxides, kaolinite, silica and gypsum.

TABLE 3. Elemental Analysis (ICP) for well X5W3 using 0.10, 0.40 and 10.0 micron filters (ppm).

Element	0.1 um	0.4 um	10 um
K	2.22	2.90	4.90
Ca	635	719	744
Na	48.4	50.3	74.0
Mg	134	152	164
Fe	0.08	0.21	0.43
Mn	65.6	72.4	74.3
Al	0.03	0.05	0.07
Ni	0.44	0.50	0.53
Sr	2.35	2.47	2.64

Air-Nitrogen

Samples were collected under nitrogen and in air to assess differences in inorganic concentrations due to oxidation effects caused by exposure of samples to atmospheric gases during sample collection and processing. Samples collected in air were exposed to O_2/CO_2 via diffusion through pump tubing and during filtration and acidification. Work by Holm et al. (28) showed that diffusion of atmospheric gases through pump tubing can introduce measurable concentration of oxygen into initially anoxic water. Significant differences (>10%) were observed in most wells, particularly those which had high iron concentrations and low pH. Variations in differences from well to well may have been caused by slightly different exposure times to air which depended mainly on water table depth, distance from well head and filtration sequence. Table 4 lists the differences in concentrations for well X3W3, where total iron was greater than 200 ppm.

TABLE 4. Elemental Analysis (ICP) for well X3W3 for samples collected in air and nitrogen (ppm, 0.40 um filter, bladder pump).

Element	air	nitrogen
Fe	177	215
Mn	37.4	44.7
Co	0.69	0.82
Cd	0.01	0.02
Cu	15.5	18.6
Ni	0.70	0.84
Zn	2.53	3.11

Similar differences were observed in wells MP1W1, X1W4 and X4W3. Data for well X4W3 are listed in Table 5. Apparently, the oxidation of samples causes the precipitation of particles and concomitant sorption of other elements, all of which are subsequently filtered out prior to acidification and preservation steps during sample processing.

TABLE 5. Elemental Analysis (ICP) for well X4W3 for samples collected in air and nitrogen (ppm, 0.40 um filter, bladder pump).

Element	air	nitrogen
Fe	6.63	9.21
Mn	34.9	41.2
Co	0.36	0.58
Cd	0.01	0.02
Cu	2.14	2.91
Ni	0.42	0.52
Zn	0.95	1.83
Pb	<.01	0.05

Additional testing is needed to determine if in-line filtration with thick walled tubing can mitigate such differences to avoid use of a cumbersome glove box in the field for collection and processing of samples.

CONCLUSIONS

A major assumption concerning assessments of colloidal mobility at the site are that the bladder pump, with its low flow rate, permits only naturally suspended particulates to be pumped to the surface. Particle size analysis techniques and scanning electron microscopy will be employed to estimate size and nature of recovered particles and test the above assumption. The results presented here are preliminary and based on a limited number of samples. While concentration differences due to different filter pore sizes were generally not striking, differences between those samples collected in air and under nitrogen were significant in wells high in iron and with low pH.

Finally, the above recommendations for sampling were practical from an operational standpoint and additional work at this and other sites is needed to address the related issues of sampling, filtration and colloidal mobility.

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DISCLAIMER

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